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“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 5438 (1977): Nitrobenzene-3-Sulphonic Acid, Sodium Salt
[PCD 9: Organic Chemicals Alcohols and Allied Products and
Dye Intermediates]



“ज्ञान से एक नये भारत का निर्माण”

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“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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IS : 5438 - 1977

Indian Standard
SPECIFICATION FOR
NITROBENZENE-3-SULPHONIC ACID,
SODIUM SALT
(*First Revision*)

UDC 547 546.269.3



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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

November 1977

AMENDMENT NO. 1 MARCH 2002
TO
IS 5438 : 1977 SPECIFICATION FOR NITROBENZENE -
3-SULPHONIC ACID, SODIUM SALT
(First Revision)

(*Page 3, clause 0.2, Structural formula*) — Insert the following below the structural formula

‘(CAS No 127-68-4)’

[*Page 4, Table 1, Sl No (ii)*] — Substitute the following for the existing

| | | | |
|-----|-----|-----|-----|
| (1) | (2) | (3) | (4) |
|-----|-----|-----|-----|

| | | | | | |
|------|---------------------------|-----------------|-----|-----|-----------------------------------|
| (ii) | Matter insoluble in water | percent by mass | Max | 0.2 | when tested as per IS 5299 : 2001 |
|------|---------------------------|-----------------|-----|-----|-----------------------------------|

(*PCD 11*)

Reprography Unit, BIS, New Delhi, India

Indian Standard
SPECIFICATION FOR
NITROBENZENE-3-SULPHONIC ACID,
SODIUM SALT
(First Revision)

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(Continued on page 2)

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(Continued from page 1)

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Panel for Sulphonic Acid Dye Intermediates, CDC 46 . P3

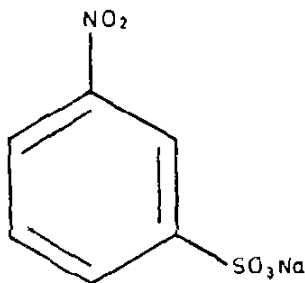
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Indian Standard
SPECIFICATION FOR
NITROBENZENE-3-SULPHONIC ACID,
SODIUM SALT
(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 15 June 1977, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Chemical Division Council.

0.2 Nitrobenzene-3-sulphonic acid, sodium salt ($C_6H_4O_3NSNa$) is an important dye intermediate used in the manufacture of metanilic acid and as a mild oxidizing agent in discharge printing of cloth. It has the following structural formula:



NITROBENZENE-3-SULPHONIC ACID, SODIUM SALT
 (Molecular Mass 225.2)

0.3 This standard was first issued in 1969. In this revised version a requirement and method of test has been prescribed for the presence of iron. Reference has also been made to IS 5299-1969*.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS: 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard

*Methods of sampling and tests for dye intermediates

†Rules for rounding off numerical values (*revised*).

IS : 5438 - 1977

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for nitrobenzene-3-sulphonic acid, sodium salt.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of white powder. It shall be free from dust and other visible impurities.

2.2 The material shall also comply with the requirements given in Table 1

TABLE 1 REQUIREMENTS FOR NITROBENZENE-3-SULPHONIC ACID, SODIUM SALT

| Sl. No | CHARACTERISTIC | REQUIREMENT (ON DRY BASIS) | METHOD OF TEST, REF TO CL No. IN APPENDIX A |
|--------|--|---------------------------------|---|
| (1) | (2) | (3) | (4) |
| i) | Assay, percent by mass (on mol mass 225.2), <i>Min</i> | 95 | A-2 |
| ii) | Solubility in water | To pass test | A-3 |
| iii) | pH of 5 percent solution | 8.5 to 10.5 | A-3 |
| iv) | Iron, ppm, <i>Max</i> | 250 | A-4 |

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in steel drums (see IS : 2552-1970*) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

3.2 Marking — Each container shall bear legibly and indelibly the following information:

- a) Name of the material;
- b) Name of the manufacturer and his recognized trade-mark, if any,
- c) Tare, net and gross mass, and
- d) Batch number.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that

*Specification for steel drums (galvanized and ungalvanized) (*first revision*).

standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. *Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution*

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 3 of IS : 5299-1969*.

4.2 Number of Tests

4.2.1 Tests for assay shall be conducted on each of the individual samples

4.2.2 Tests for the determination of remaining characteristics, namely solubility in water, pH of solution and iron shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The lot shall be declared as conforming to the requirement of assay if each individual test result satisfies the relevant requirement given in Table 1.

4.3.2 *For Composite Sample* — For declaring the conformity of the lot to the requirements of the other characteristics tested on the composite sample, the test results for the characteristics shall satisfy the relevant requirements given in Table 1.

5. TEST METHODS

5.1 Tests shall be carried out as prescribed in Appendix A. Reference to relevant clauses of Appendix A is given in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

NOTE — ‘ Pure chemicals ’ shall mean chemicals that do not contain impurities which affect the results of analysis.

*Methods of sampling and tests for dye intermediates.

†Specification for water for general laboratory use (*second revision*).

APPENDIX A
(*Table 1, and Clause 5.1*)

**METHODS OF TEST FOR NITROBENZENE-3-SULPHONIC
ACID, SODIUM SALT**

A-1. PREPARATION OF SAMPLE

A-1.1 Dry the material at $105^{\circ} \pm 1^{\circ}\text{C}$ to constant mass. Grind and mix well. Transfer the material to a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this *prepared sample* for tests.

A-2. ASSAY

A-2.1 Test the material for assay as prescribed in **14** of IS : 5299-1969*.

A-3. DETERMINATION OF SOLUBILITY IN WATER AND pH OF SOLUTION

A-3.1 Weigh 5.0 g of the *prepared sample* (see **A-1.1**) and dissolve it in about 80 ml of water in a 250-ml beaker. Transfer the solution to a 100-ml graduated flask and make the volume up to the mark. Shake well. The solution shall be clear without any suspended matter. Measure the pH of the solution by a standard pH meter or by another means of equivalent accuracy.

A-4. DETERMINATION OF IRON CONTENT

A-4.1 Outline of the Method — Thioglycolic acid gives a reddish-violet colouration with ferrous and ferric ions in an alkaline medium. The ash of the material is dissolved in hydrochloric acid, thioglycolic acid added and then made alkaline by the addition of ammonia. The colour developed is compared with that obtained with standard iron solution similarly treated.

A-4.2 Apparatus

A-4.2.1 Volumetric Flasks — two, one mark, 1 000 ml capacity.

A-4.2.2 Graduated Flask — one mark, 25 ml capacity.

A-4.2.3 Nessler Cylinders — two, 100 ml capacity.

*Methods of sampling and tests for dye intermediates.

A-4.3 Reagents

A-4.3.1 Hydrochloric Acid — relative density 1.16.

A-4.3.2 Ammonium Hydroxide — relative density 0.90.

A-4.3.3 Thioglycolic Acid Solution — 10 percent (v/v).

A-4.3.4 Citric Acid Solution — 30 percent (m/v).

A-4.3.5 Standard Iron Solution — Dissolve 0.7022 g of ammonium ferrous sulphate [$(\text{NH}_4)_2 \text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}$] in 50 ml of dilute sulphuric acid and transfer to one of the 1 000-ml volumetric flasks. Dilute with water to 1 000-ml mark and mix well. Pipette out 100 ml of this solution into the second 1 000-ml volumetric flask. Dilute again to 1 000 ml. One millilitre of this solution contains 10 μg of iron.

A-4.4 Procedure — Weigh accurately about 5 g of the material into a platinum or silica basin. Heat gently at first to remove as much volatile matter as possible and then ignite at dull red heat. If carbon is not removed, easily allow the dish to cool with its contents. Moisten the carbon matter with concentrated nitric acid and heat again strongly until all the carbon is removed. Cool and dissolve the ash in about 15 ml of hydrochloric acid. Heat the basin on a boiling water-bath agitating with a stirrer of platinum wire until all the residue has dissolved. Allow to cool, transfer to the 25-ml volumetric flask, dilute to mark with water and mix thoroughly. For each determination transfer 10 ml of this solution to a 100 ml Nessler cylinder, dilute to about 30 ml and add 0.5 ml of the citric acid solution followed by 1 ml of thioglycolic acid solution. Add ammonium hydroxide carefully until a reddish-purple colour just appears and then add 0.5 ml in excess. Dilute to 100 ml and mix thoroughly. To about 90 ml of water in the second 100-ml Nessler cylinder add 2 ml of concentrated hydrochloric acid, 0.5 ml of citric acid solution followed by 1 ml of thioglycolic acid solution and 3 ml of ammonium hydroxide. Add the standard iron solution slowly from a burette shaking with each addition until the depth of colour in the two Nessler cylinders is identical when they are viewed along their axis. Record the volume of standard iron solution added.

A-4.5 Calculation

$$\text{Iron content (as Fe) ppm} = \frac{V \times 25}{M}$$

where

V = volume in ml of standard iron solution used, and

M = mass in g of the material taken for the test,

INDIAN STANDARDS
ON
SULPHONIC ACID DYE INTERMEDIATES

IS ·

| | |
|-----------|--|
| 2740-1973 | Sulphanilic acid, technical (<i>first revision</i>) |
| 3229-1973 | Naphthionic acid (sodium salt) (<i>first revision</i>) |
| 4265-1975 | 4, 4'-Diaminostilbene-2, 2'-disulphonic acid (<i>first revision</i>) |
| 4425-1967 | <i>p</i> -Nitrotoluene- <i>o</i> -sulphonic acid |
| 4528-1977 | 4, 4'-Dinitrostilbene-2, 2'-disulphonic acid (disodium salt) (<i>first revision</i>) |
| 5045-1976 | Metanilic acid, technical (<i>first revision</i>) |
| 5299-1969 | Methods of sampling and tests for dye intermediates |
| 5438-1977 | Nitrobenzene-3-sulphonic acid, sodium salt (<i>first revision</i>) |
| 6264-1971 | J-acid |
| 7362-1971 | Tobias acid |
| 7645-1975 | Phenyl J-acid, technical |
| 7646-1975 | Benzoyl J-acid, technical |
| 7704-1975 | 4-Aminotoluene-3-sulphonic acid |
| 8059-1976 | 2-Chloro-5-aminotoluene-4-sulphonic acid |
| 8120-1976 | Schaeffer's acid (sodium salt), technical |
| 8233-1976 | Phenyl <i>p</i> <i>n</i> acid, technical |

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

| Quantity | Unit | Symbol |
|---------------------------|----------|--------|
| Length | metre | m |
| Mass | kilogram | kg |
| Time | second | s |
| Electric current | ampere | A |
| Thermodynamic temperature | kelvin | K |
| Luminous intensity | candela | cd |
| Amount of substance | mole | mol |

Supplementary Units

| Quantity | Unit | Symbol |
|-------------|-----------|--------|
| Plane angle | radian | rad |
| Solid angle | steradian | sr |

Derived Units

| Quantity | Unit | Symbol | Conversion |
|----------------------|---------|--------|---------------------------------|
| Force | newton | N | 1 N = 1 kg.m/s ² |
| Energy | joule | J | 1 J = 1 N.m |
| Power | watt | W | 1 W = 1 J/s |
| Flux | weber | Wb | 1 Wb = 1 V.s |
| Flux density | tesla | T | 1 T = 1 Wb/m ² |
| Frequency | hertz | Hz | 1 Hz = 1 c/s (s ⁻¹) |
| Electric conductance | siemens | S | 1 S = 1 A/V |
| Pressure, stress | pascal | Pa | 1 Pa = 1 N/m ² |

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